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SIXTH QUARTERLY REPORT

INVESTIGATION OF ELECTRODE MATERIALS FOR ALKALINE BATTERIES

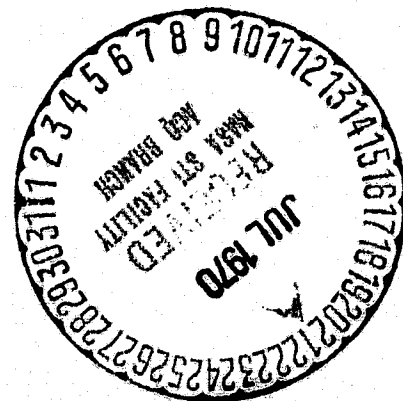
JPL 952265

This work was performed for the Jet Propulsion Laboratory,
California Institute of Technology, sponsored by the National
Aeronautics and Space Administration under Contract NAS7-100.

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Idaho State University
Pocatello, Idaho
November 25, 1969

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ABSTRACT

As a first approximation, the diffusion coefficient for the silver species in 9.94 VF KOH has been found to be $2.75 \pm .70 \times 10^{-6} \text{ cm}^2 \text{ sec}^{-1}$ at $25^\circ \pm 0.1^\circ$. Work is continuing to improve this value and to determine values for other conditions.

Argentite oxide has been found to decompose slowly at temperatures at least as low as 86° and possibly lower. Both AgO and Ag₂O can be activated to take up a substance or substances from the atmosphere at room temperature. Activation temperatures at least as low as 66° are effective. No adsorption occurs without prior heating. The substances most likely to be adsorbed are water and oxygen. Weight increases after temperature activation exceed weight losses during heating.

Amalgam electrodes in propylene carbonate and in dimethyl formamide do not show good high-rate discharge characteristics. In general, maximum discharge rates did not exceed 5 mA which would correspond to about 1 mA/cm^2 . Lithium amalgams in LiClO₄ produced open-circuit voltages around -2.1 V vs the Hg/HgO electrode.

A secondary cell, K(Hg)/10 VF KOH//CuCl₂, DMF/Cu, produced an open-circuit voltage of 2.13 V. Discharge at 10 mA yielded almost 100% charge recovery. The IR drop was excessive at about 1 V.

When a K(Hg) electrode is discharged at $2,688 \text{ mA/cm}^2$, recovery is low when the total charge is low. Recovery ranges from 29% at a total charge of 1 mA-hr/g Hg to 90% at 9 mA-hr/g Hg. In the same range, open-circuit voltages change from -1.62 V to -1.76 V vs Hg/HgO.

Steady-state self-discharge rates of K(Hg) electrodes into saturated KOH average about 0.071 $\mu\text{equiv/hr}$ indicating complete discharge in about 1½ years.

The objectives of the contract are four-fold:

- (1) Study of the reduction of Ag(I) by zinc.
- (2) Study of the thermal decomposition of AgO and Ag₂O.
- (3) Study of amalgam electrodes.
- (4) Study of the evolution of gas at electrodes.

This report will be limited to the first three objectives.

A. DEPOSITION OF SILVER ON ZINC FROM KOH SOLUTIONS.

Experimental

Some diffusion measurements were made using the method of Anderson and Saddington¹. The capillary diffusion-cell was modified from that described previously². No cement was found which would hold the cover-glass bottoms securely in the presence of concentrated KOH. Therefore, the cells were constructed by carefully sealing capillary tubing in a flame and flattening the end while it was still hot. The bottom of the capillary section appears to be flat as required for this technique.

Calculation of the diffusion constant by this method requires that the cell length be known rather than its volume. The length was measured by inserting a straight piece of wire, cutting it and carefully grinding it flush with the open end of the cell. The wire was removed and its length measured with a micrometer. It is a minor disadvantage that the cells are not of uniform length.

The main cell chamber was filled with 40 ml of 9.94 VF KOH saturated with Ag₂O. The diffusion cell was filled with 9.94 VF KOH saturated with Ag₂O which contained some ^{110m}Ag as tracer. The capillary cell was mounted in the main cell chamber with the open end above the surface of

the inactive solution. The complete unit was installed in a water bath controlled at $25^{\circ} \pm 0.1^{\circ}$. The system was allowed one hour for temperature equilibration whereupon the capillary cell was gently lowered to the bottom of the main chamber. The system was allowed to stand undisturbed for about five days, then the capillary was removed, the contents washed into the counting vials containing scintillator fluid³ and the samples counted. Exactly the same amount of active stock solution was counted.

Samples were counted with a Nuclear-Chicago Model 703 liquid scintillation system which has been described previously³. Sample vials were held at 50° F in the sample chamber.

Results and Conclusions

The equations used to calculate the diffusion coefficient were taken from Anderson and Saddington¹:

$$\gamma = (8/\pi^2) \exp(-\theta) \quad (1)$$

$$\theta = (\pi^2 D \lambda) / 4l^2 \quad (2)$$

where γ = fraction of original activity remaining in the diffusion cell

D = the diffusion constant for the Ag(I) species

λ = diffusion time in seconds

l = length of diffusion cell in cm.

The average value of D for six runs was $2.75 \pm .70 \times 10^{-6} \text{ cm}^2 \text{ sec}^{-1}$.

The precision is poor, but some experimental problems are being investigated and it is hoped that better results will be forthcoming.

Proposed Work

Diffusion measurements will be continued at the same and at different KOH concentrations. The scintillator "cocktail" will be changed

to provide a gel-like suspension in order to eliminate the problems which apparently are arising from inhomogeneity in the counting vials.

B. THERMAL DECOMPOSITION OF AgO AND Ag_2O

Experimental

AgO , obtained from Ames Chemical Works, and Ag_2O , obtained from K & K Laboratories, were used without further purification.

For the analytical procedure, initial heating ("drying") was done in a Labline drying oven at 105° . Thermal decompositions were carried out in a Tempco Model 173B muffle furnace set at 230° for the AgO - Ag_2O transition and at 400° for the Ag_2O - Ag transition.

Thermogravimetric and "adsorption" measurements were made with a Cahn Model RG electrobalance mounted in a Cahn #2005 Glass Vacuum Bottle accessory. The sample was contained in a platinum micro-pan suspended in a 10 mm diameter hangdown tube and was heated with a Lindberg Type 123-2 8" tube furnace. Several layers of sheet aluminum were placed on top of the furnace to shield the balance from furnace heat. The hangdown tube and all port caps were coated with platinum and grounded to provide electrostatic shielding. Temperature was controlled with an F & M Model 240M temperature controller. Temperature was recorded with a Texas Instruments Recti/Riter II 1-mV recorder operated through a Cahn Recorder Controller. Weight changes were monitored with a Dohrmann Model RSC-1100 recorder.

Results and Discussion

It was previously reported that a thermal method of analysis for Ag , Ag_2O , and AgO seemed feasible. The sample was to be dried at about 100° initially where no significant decomposition was expected.

The cold weight at this point would be considered the sample weight. It was then to be heated at 230° for complete decomposition to Ag_2O after which it would be cooled and weighed. The weight loss at this point would correspond to the amount of AgO present in the sample. Finally, the sample was to be heated at 400° for complete decomposition to Ag , cooled and weighed. From this information, it should be possible to calculate the amounts of each species present in the original sample.

The results of the experiments performed according to the above plan were not satisfactory. The "drying" at 100° suggested that something more might be occurring because constant weight was not obtainable and because the "dried" samples gained weight on standing in a calcium chloride desiccator. While this gain did not amount to more than a few milligrams, the uncertainty amounted to several percent in the major component and as much as 10% in the minor component. While the gain took place over several days, it left an uncertainty as to what exactly was happening. Similar phenomena were observed with both AgO and Ag_2O .

Errors in all of the analyses were high and led to calculated total amounts of original sample 6-10% greater than was actually present. Obviously, something unexpected was happening.

A thermogram was begun at the very slow nominal heating rate of $0.5^{\circ}/\text{min}$ to try to detect any obvious breaks in the weight-temperature curve which would indicate what was being lost and at what temperature. Heating was started after equilibration at 35° and continued for four hours the first day. The temperature was increased to about 100° in this time with some small loss in weight as expected. Loss of moisture, at least, would be anticipated at this temperature. The heat was turned off over night and the system remained at virtual room temperature for

about 16 hours. During this period, the sample regained all of the weight it lost during the heating and 65 μ g over its original weight.

In a 28 mg sample, this amounts of 0.3%. While this is not a great deal, when it represents a gain over the initial sample weight, it becomes significant.

In order to be sure than the gain was not simply an instrument drift or a moisture absorption on balance components and sample pan, an aluminum weight was placed in the pan and left at room temperature for 18 hours; no weight change was observed. A further experiment was made to see if a powdered material would absorb moisture at room temperature. Ames Chemical Works silver Type SG powder of 99.9+ purity having an average particle size of 1.45 micron was left on the sample pan at room temperature for seven hours; no weight change was observed. In this case, the initial weight was 23.650 mg. This sample was then heated at 106⁰ for one hour and then allowed to stand at room temperature for 15 hours; no weight change was observed. The sample was heated again at the same temperature for six hours and then allowed to stand at room temperature for 18 hours; again, no weight change was observed. Obviously, silver powder did not behave as its oxides.

Evidently, the weight-gain phenomenon is a characteristic of the oxides of silver. Thus, it seemed likely that Ag₂O would show a slow weight increase if left at room temperature without prior heating. A sample weighting 24.685 mg was placed in the balance pan and left at room temperature for 18½ hours; no change in weight was observed. The temperature was raised to 66⁰ for about 95 hours, the heat was turned off and the system left for about 50 hours at room temperature, the sample was then heated at 76⁰ for about 30 hours and, again, the heat was turned

off. The current results are shown in Figure 1. The experiment is continuing.

It cannot be said that the weight loss at 66° is anything other than moisture loss, since it amounts to less than 0.3% in about 4 days. However, there is no way of knowing from the available data whether the weight will ultimately level off at some reasonable value. The rapid increase at room temperature after the initial heating is striking and amounts to 0.75% in two days. The decrease at 76° is not surprising now, although the rate is about six times greater than that at 66° . The upward trend at room temperature appears to be quite similar to the previous increase.

No serious explanation for these observations is offered at this time. Much more work is necessary. It seems reasonable to suppose that heating the oxide, even at the comparatively low temperature of 66° , causes a change in surface structure allowing a fairly rapid adsorption. Water is the obvious absorbent, yet this is hard to justify because the oxide is relatively isolated from the laboratory atmosphere, although the system is not gas-tight. Oxygen may also be the adsorbent. One must recall the gassing phenomenon on heat-pressed AgO electrodes observed by Butler⁴. At any rate, the problem needs further investigation.

Proposed Work

Thermograms of the type described above will be continued with both AgO and Ag₂O. An attempt will be made to determine the maximum relative adsorption by allowing the system to stand after activation until the weight levels off. The minimum temperature required for activation will be determined as well as the minimum temperature at which true decomposition

occurs. Similar experiments will be conducted in vacuum, in oxygen-free nitrogen, in dry air, and in pure oxygen. An attempt will be made to identify the phenomenon and describe its mechanism.

C. AMALGAM ELECTRODES

Several amalgam electrodes show promise as rechargeable anodes for galvanic cells. These are all alkali-metal amalgams in very concentrated aqueous solutions. Most of the electrodes show a fairly high self-discharge rate. The only one that seems to have a reasonable shelf-life is the $K(Hg)/KOH(satd)$ electrode. Therefore, an investigation of other solvent and electrolyte systems which might retain the advantages of the amalgam electrode performance while showing improved stand-life appears reasonable. Of the various non-aqueous solvents reported in the literature for battery electrolytes, the most promising seem to be dimethyl formamide (DMF), propylene carbonate (PC), dimethyl sulfite, and acetonitrile. Two of these solvents were tried and the results are reported.

Work on discharge rates and stand life is also continuing. The results are brought up to date.

Experimental

The method for measuring high discharge rates with a hanging-drop mercury electrode has been described². This work was continued to determine the effect of total charge on recovery and open-circuit voltage and to determine the resistive (IR) voltage drop in the electrolyte and electrode-electrolyte solution interface during very high discharge rates.

Gas evolution experiments using Warburg equipment is being continued. The method has been described.

Stand-life experiments are being continued; these have been described previously³. They consist of charging an electrode to some pre-determined value, transferring the amalgam to a separate tube containing fresh electrolyte solution, and letting the system stand at room temperature for a selected time. The amalgam is then transferred back to a cell and discharged. The percent recovery of charge gives an indication of stand-life.

Experiments with different solvent systems were performed in simple containers equipped with a cup to hold the amalgam. Contact with the amalgam was made with a platinum wire sealed through the bottom of the cup. In some cases, the containers were connected by a crossarm to make an H-cell. Counter electrodes were usually platinum wire although a different arrangement was used occasionally. When different electrolyte solutions were used in the same cell, a polarographic H-cell containing a sintered-glass separator in the crossarm was employed. Reference electrodes were Hg/HgO, 20% KOH.

The non-aqueous electrode systems were usually discharged through a constant-current power supply; occasionally, they were discharged through a decade resistor.

Results and Discussion

Polypropylene carbonate (PC) shows little promise as a solvent for electrolytes which would be useful with amalgam electrodes. KHCO_3 , KOH, KCl, and potassium acid phthalate ($\text{KHC}_8\text{H}_4\text{O}_4$) were only slightly soluble in PC while maximum current-densities obtainable were less than 100 μA . The alkali-metal perchlorates are more soluble in PC and showed better operating characteristics. These are shown in Table 1. Even so, they do not perform at a level desired for amalgam electrodes.

Dimethyl formamide (DMF) is little better as a solvent. Results from several alkali-metal perchlorates are shown in Table 2. Output currents are low and, except for the Li(Hg) electrode, open-circuit voltages are no better than those found in aqueous systems.

A complete cell employing a Li(Hg)/LiClO₄ - DMF anode and a Cu(Hg)/CuCl₂·2H₂O - DMF cathode accepted a charge of 2.5 mA-hr and produced an open-circuit voltage (E_{oc}) of 2.25 V. However, it yielded a maximum discharge rate of 2 mA with only 60% charge recovery.

A cell employing a Na(Hg)/NaClO₄ - DMF anode and a Cu/CuCl₂ - DMF cathode accepted a charge of 3.3 mA-hr and yielded an E_{oc} = 2 V. The Cu anode was a wire. When discharged through a 300-ohm resistor, a steady current of 2 mA was produced until 98% of the charge was recovered. This is not exceptional performance and the system would be of little advantage unless it should show phenomenal cycle life.

An H-cell construction was adopted to make a cell containing a K(Hg)/10 VF KOH//CuCl₂ - DMF/Cu(wire) system. The cell showed an intolerably high resistance which is not surprising since a copper oxide or hydroxide formed at the sintered-glass separator almost immediately. The short-circuit current for the cell was 10mA.

A similar cell was constructed, the only difference being that a 46 cm² copper plate was used in place of the copper wire. This cell was charged at 50 mA for 6 min where it produced E_{oc} = 2.13 V. The cell was discharged through a decade resistor. With $R = 0$, the current was 20 mA. With $R = 140$ ohms, a nominal current of 10 mA was maintained at about 1.1 V for 35 min. At the end, the cell voltage dropped abruptly to 0.5 V. The charge recovery is calculated to be somewhat greater than 100%. The charge rate and, hence, the total charge is quite accurately

known since charging is accomplished with a calibrated constant-current supply. The discharge current was measured with a simple VOM and was observed to fluctuate considerably making the total energy release on discharge difficult to determine. Nevertheless, good recovery is evident. A better cell design might provide better current-voltage characteristics. The cell operated at a voltage about one volt below the open-circuit value; this was probably largely IR drop.

None of the cells tried so far has been very satisfactory. It appears that aqueous systems are much more satisfactory and the major emphasis will be in that direction in the future.

The effect of total charge on open-circuit voltage and charge recovery is of interest. A hanging-drop K(Hg) electrode was operated in saturated aqueous KOH at a discharge rate of $2,688 \text{ mA/cm}^2$. A fresh drop was used for each experiment. The total charge was varied and the E_{oc} and percent recovery determined for each case. The results are shown in Table 3. It can be seen that the recovery is poor at the low concentrations and increases fairly steadily with increasing concentration. However, the recovery seems to stabilize at 4 or 5 mA-hr/g Hg with only a slow increase up to 9 mA-hr/g Hg. The E_{oc} also decreases steadily with increasing amalgam concentration. This decrease is not surprising but the magnitude of the change is. If the Nernst equation were valid, a maximum change of 59 mV would be expected as compared with the observed change of 140 mV. There should be no effect from non-ideality in the aqueous phase because its composition remains constant. Thus, non-ideal behavior in the amalgam is indicated. The loss of recovery at low total charge is not explained. One would expect the effective recovery to decrease during a given discharge as the remaining charge decreased. This does not occur, apparently.

Figure 2 shows the effect of discharge rate on IR drop for a hanging drop system in which the tip of the Luggin capillary is positioned about 0.5 cm from the drop. All drops were charged to 9.1 mA-hr/g. The linear relationship between current-density and voltage drop suggests that it is, indeed, a resistive effect. The point at 8.15 A/cm^2 may be in error since it represents a single measurement while the others represent duplicate measurements and because discharge times are very short at this current-density and the high IR drop is difficult to measure accurately. This experiment was conducted to verify that the low voltages observed during high discharge rates are probably not electrode-reaction effects.

Warburg experiments on the gas evolution from amalgams are still unsatisfactory. The lack of reproducibility evident in Figure 3 is disturbing. Since the oxidation of the potassium in the amalgam is very slow, one can assume that its activity is changing very slowly. Thus the curvature observed in the early part of the curves must be attributed to slow diffusion of the active material to the electrode surface. If diffusion is the controlling factor, a steady state should be reached at which the oxidation rate would be a constant over a considerable period. This has generally been the case as is indicated by the constant slopes over the major portion of each experiment. The differences in slope from one experiment to another are not expected. Two slopes agree fairly well at $0.046 \mu \text{ equiv./hr}$ and $0.050 \mu \text{ equiv/hr}$ while the third is further off at $0.078 \mu \text{ equiv/hr}$ and the fourth is considerably higher at $0.11 \mu \text{ equiv/hr}$. It is hoped that future experiments will result in better reproducibility. The average self-discharge rate into the saturated KOH solutions is $0.071 \pm 0.035 \mu \text{ equiv/hr}$.

At this average rate, 548 days would be required for complete discharge of the electrode.

Only one run has been made using a 9.9 VF KOH solution. The results are shown in Figure 4. An essentially constant slope of 1.61 μ equiv/hr develops quite quickly. At this average rate, the electrode could be expected to discharge completely in about 24 days. This corresponds fairly well with the results shown in Table 4 for conventional stand-life measurements.

The results of stand-life experiments are shown in Tables 4 and 5. The K(Hg) electrode in 10 VF KOH has a relatively poor stand-life, discharging completely in periods ranging from 25 to 60 days. None of these retained more than 50% charge for more than about 300 hours. This behavior would be expected after considering the data from the Warburg experiments.

The K(Hg) electrode in saturated KOH behaves much better. The loss is only about 6% over a period of about 75 days. Relatively few experiments have been made in the saturated electrolyte because of the long times involved. It is evident that concentrations must approach saturation if the electrodes are to be kept for any length of time.

Proposed Work

Work on stand-life and self-discharge rates will be continued. The effects of surface area and stirring on the latter will be considered.

Careful measurement of open-circuit potentials have begun and will continue. The effect of amalgam and electrolyte concentrations will be determined. Drift and temperature effects will be investigated.

Attempts to find a suitable cathode for use with the amalgam anodes will be continued.

Table 1

Amalgam Electrodes in Propylene Carbonate Solutions

Hg/HgO electrode E = -0.924 V vs standard H₂ electrode

<u>Amalgam</u>	<u>Electrolyte</u>	<u>Charge (mA-hr)</u>	<u>E_{oc} vs Hg/HgO (V)</u>	<u>Discharge (mA)</u>	<u>Recovery (%)</u>
Li(Hg)	LiClO ₄	7.5	-2.0	5	75
Li(Hg)	LiClO ₄	1.5	-2.08	5	20*
				1	66*
Na(Hg)	NaClO ₄ (satd)	3.3	-1.75	5	45*
				2	66*
K(Hg)	KClO ₄ (satd)	5.0	-1.86	2	----

* Total recovery of an electrode after discharge at the indicated rate.

Table 2

Amalgam Electrodes in Dimethyl Formamide Solutions

Hg/HgO electrode $E = -0.924$ V vs standard H_2 electrode.

<u>Amalgam</u>	<u>Electrolyte</u>	<u>Charge (mA-hr)</u>	<u>E_{oc} vs Hg/HgO (V)</u>	<u>Discharge (mA)</u>	<u>Recovery (%)</u>
Li(Hg)	LiClO ₄	5.0	-2.25	5	75
Na(Hg)	NaClO ₄	4.0	-1.70	5	96
K(Hg)	KClO ₄ (satd)	6.7	-1.90	5	9*
				2	9.1*
				1	24.8*
				2	45.7*
				5	88

* Total recovery of an electrode after discharge at the indicated rate.

Table 3

Effect of Amalgam Concentration on Open-Circuit Voltage and Recovery

K(Hg) electrode. Discharge rate = $2,688 \text{ mA/cm}^2$. Hg/HgO electrode

$E = -0.924 \text{ V}$ vs standard H_2 electrode.

<u>Concentration</u> <u>(mA-hr/g Hg)</u>	<u>E_{oc} vs Hg/HgO</u> <u>(V)</u>	<u>Recovery</u> <u>(%)</u>
1	-1.62	29
2	-1.64	61
3	-1.66	74
4	-1.69	85
5	-1.70	85
6	-1.73	87
7	-1.74	88
8	-1.75	89
9	-1.76	90

Table 4

Stand-Life of K(Hg) Electrodes in 10 VF KOH

E_{oc} is the measured value at the end of the stand time and before discharge. Discharge rate = 50mA. Each value represents an individual electrode.

<u>Charge (mA-hr)</u>	<u>Stand Time (hours)</u>	<u>E_{oc} vs Hg/HgO (V)</u>	<u>Recovery (%)</u>
68	0.17	-1.68	81
	35	-1.70	84
	157	-1.67	68
	302	-1.67	43
	455	-1.59	0
101	0.08	-1.73	96
	311	-1.67	25
	312	-1.50	0
	312	-1.60	2
105	1.15	-1.69	54
	1348	-1.54	0
	1348	-1.69	47
	1561	-1.66	15
105	0.5	-1.67	21
	1370	-1.00	0
	1371	-1.64	7
	1371	-1.67	16

Table 5

Stand-Life of K(Hg) Electrodes in Saturated KOH

<u>Charge (mA-hr)</u>	<u>Stand Time (hours)</u>	<u>E_{oc} vs Hg/HgO (V)</u>	<u>Recovery (%)</u>
105	768	-1.68	98
	1802	-1.64	94

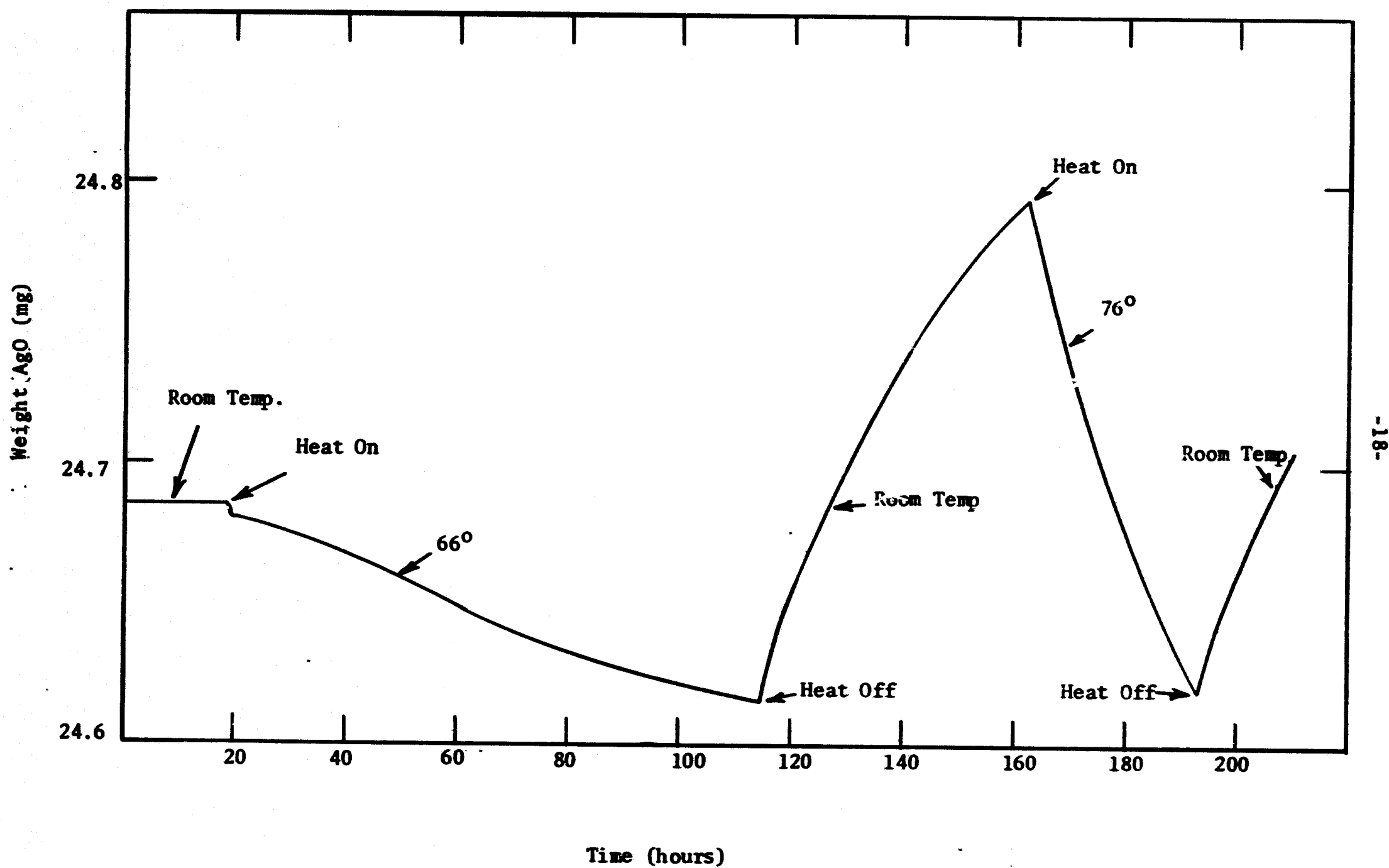


Figure 1. Adsorption and Desorption on AgO

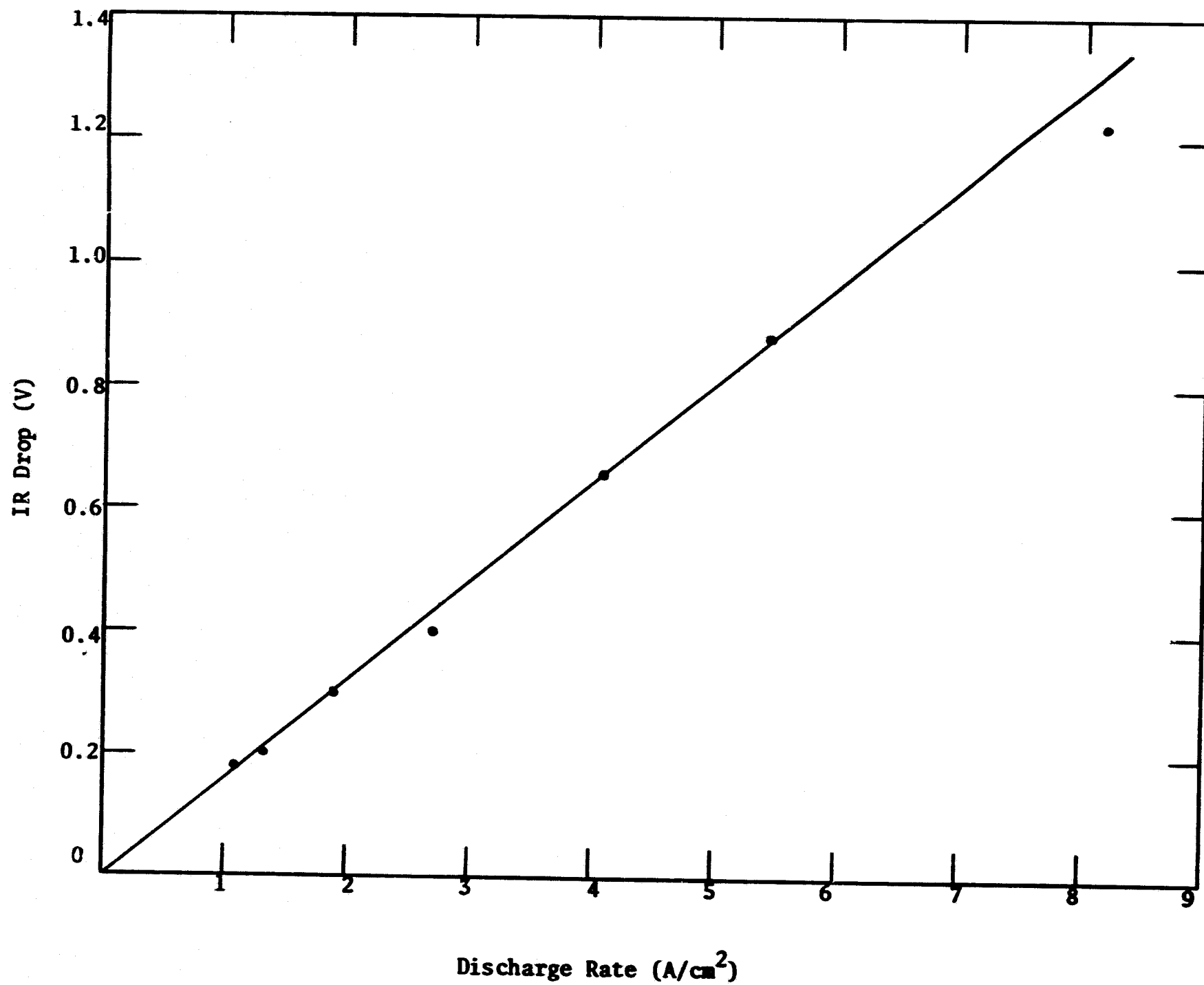
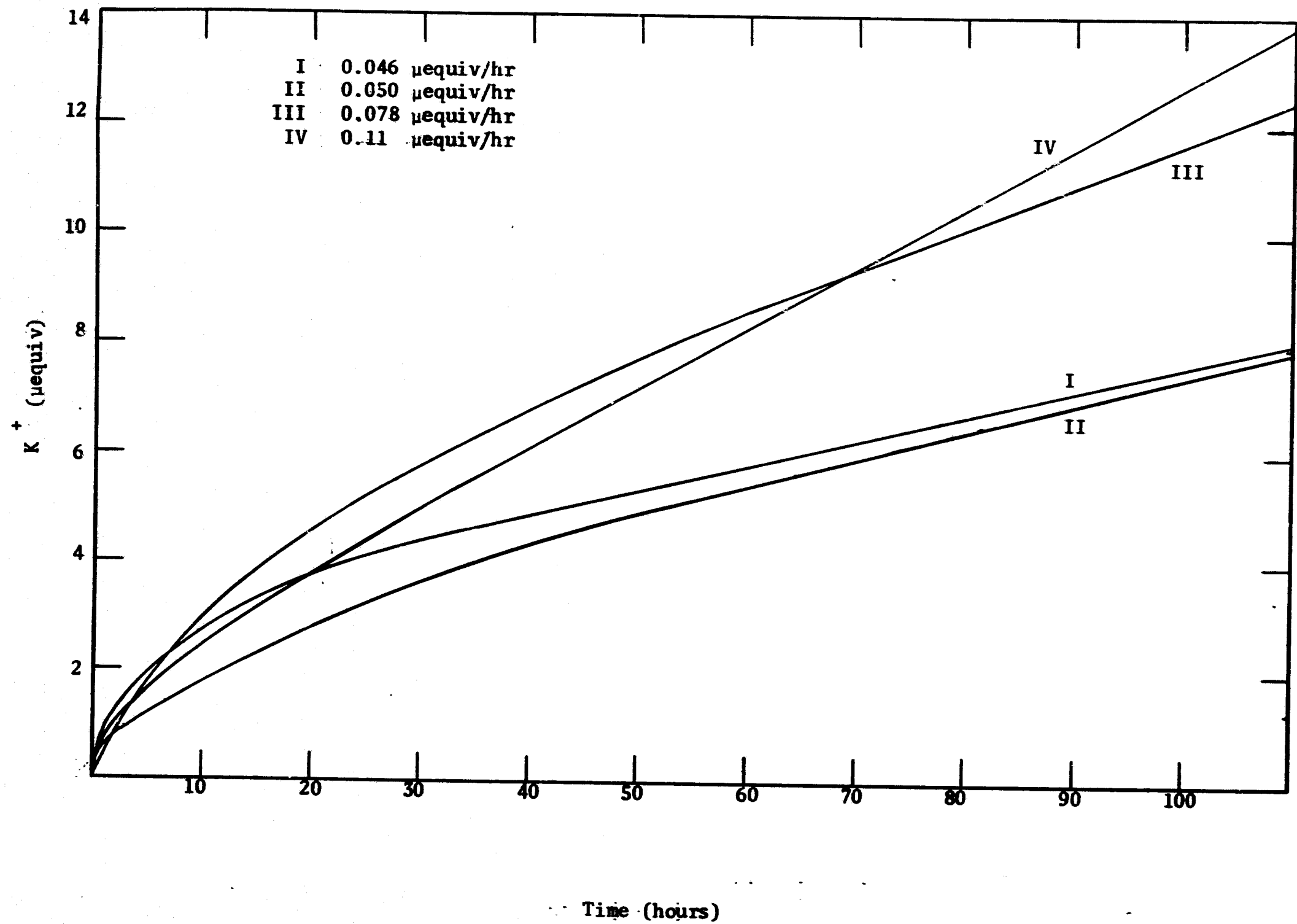


Figure 2. Effect of Discharge Rate on IR Drop for Amalgam Electrodes



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Figure 3. Self-Discharge Rate of K(Hg) in Saturated KOH Solutions

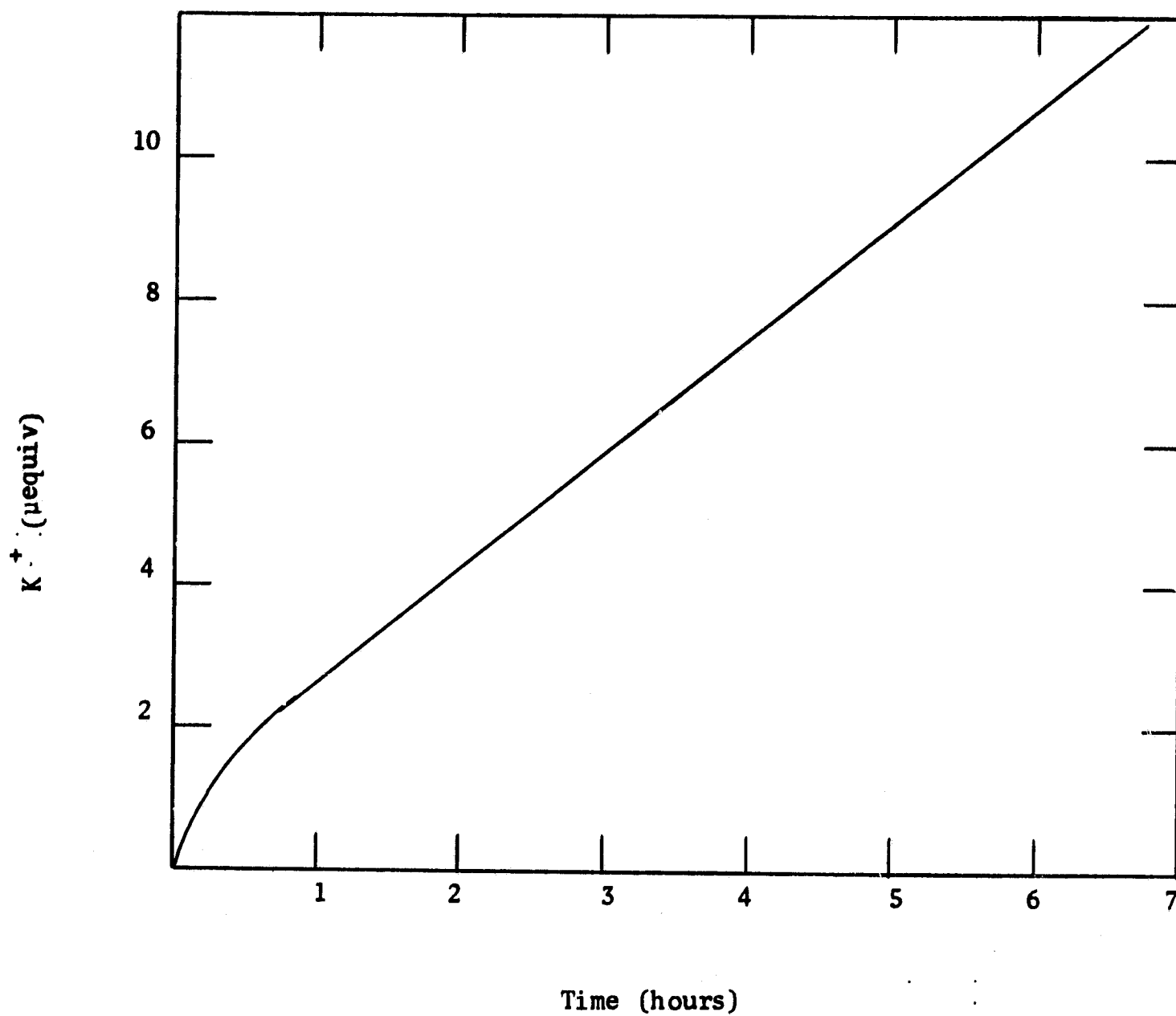


Figure 4. Self-Discharge Rate of K(Hg) in 10 VF KOH

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